PCT/GB 2004/ 005273

INVESTOR IN PEOPLE

The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

Signed Stephen Hordley

Dated 6 January 2005

tents Form 1/77

nts Act 1977
le 16)

THE PATENT OFFICE
C
19 DEC 2003
RULE 97
NEWPORT

220EC03 E861022-1 D02903
P01/7700 0.00-0329529.2 ACCOUNT CHA

1/77

The Patent Office

Cardiff Road
Newport
South Wales
NP10 8QQ

equest for grant of a patent

e the notes on the back of this form. You can also get explanatory leaflet from the Patent Office to help you fill in is form)

1. Your reference — 11377P4 GB/CMB

0329529.2

2. Patent application number
(The Patent Office will fill this part in) — 19 DEC 2003

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Reckitt Benckiser N.V.
Kantoorgebouw De Appelaer
De Fruittuinen 2-12
2132 NZ Hoofddorp
Netherlands
07921075005

Patents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation — Netherlands

4. Title of the invention — Process

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

Craig M Bowers
Group Patents Department
Reckitt Benckiser plc
Dansom Lane
Hull
HU8 7DS
United Kingdom

Patents ADP number *(if you know it)* — 07799521001

6. Priority: Complete this section if you are declaring priority from one or more earlier patent applications, filed in the last 12 months.

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. Divisionals, etc: Complete this section only if this application is a divisional application or resulted from an entitlement dispute (see note f)

Number of earlier UK application	Date of filing *(day / month / year)*

8. Is a Patents Form 7/77 (Statement of inventorship and of right to grant of a patent) required in support of this request? — Yes

Answer YES if:

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.

Otherwise answer NO (See note d)

Patents Form 1/77

9. Accompanying documents: A patent application must include a description of the invention. Not counting duplicates, please enter the number of pages of each item accompanying this form:

Continuation sheets of this form	
Description	11
Claim(s)	3
Abstract	1
Drawing(s)	0

10. If you are also filing any of the following, state how many against each item.

Priority documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent (Patents Form 7/77)	
Request for a preliminary examination and search (Patents Form 9/77)	One
Request for a substantive examination (Patents Form 10/77)	One
Any other documents (please specify)	FS1

11. I/We request the grant of a patent on the basis of this application.

Signature(s) Craig M Bowers Date 16 December 20(

12. Name, daytime telephone number and e-mail address, if any, of person to contact in the United Kingdom

Craig M Bowers

01482 583730 Craig.Bowers@ReckittBenckiser.com

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 08459 500505.

b) Write your answers in capital letters using black ink or you may type them.

c) If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.

d) If you have answered YES in part 8, a Patents Form 7/77 will need to be filed.

e) Once you have filled in the form you must remember to sign and date it.

f) Part 7 should only be completed when a divisional application is being made under section 15(4), or when an application is being made under section 8(3), 12(6) or 37(4) following an entitlement dispute. By completing part 7 you are requesting that this application takes the same filing date as an earlier UK application. If you want the new application to have the same priority date(s) as the earlier UK application, you should also complete part 6 with the priority details.

PROCESS

The present invention relates to a process for making pellets of a thermoplastic extrudable polymer.

Processes for making pellets of thermoplastic extrudable polymer are well known in the plastic industry. Typically the pellets are cylindrical and approximately 3mm in diameter and 3mm in length. The pellets are used in a wide range of plastic article manufacturing processes.

The pellet manufacturing process generally includes a plastification step. In this step the formulation to be pelletised is melted and fed into a twin screw extruder. This has been seen to be beneficial as the pellets produced have been found to comprise of a homogeneous blend of the pellet components due to effective mixing of all molten components in the extruder.

EP-A-0 415 357 describes the making of pellets comprising polyvinylalcohol (PVOH) by melt extrusion with the extrusion being carried out in the temperature range of 150-195°C.

Pelletising processes having a plastification step have several disadvantages associated therewith. The principle disadvantage is the requirement for heating, which means that the energy consumption of these processes is very high.

Furthermore these 'hot' processes are not suitable for polymers which are heat sensitive (such as PVOH) due to heat induced decomposition. Also these 'hot' processes give a

heat history to the polymer which has been found to negatively influence properties of the polymer. In the case of PVOH this has been found to detrimentally affect the PVOH water solubility .

In other pelletising processes dry compaction of the pellet components is carried out at low temperature. Thus the disadvantages of the 'hot' processes are avoided.

WO-A-98/26911 describes a low temperature process for the manufacture of PVOH pellets. In the process the pellets components, in this case a mixture of powdered PVOH and various additives such as plasticisers is fed between two rollers and compressed into pellets. The PVOH component in the pellet blend is not melted in the process and so the issue of heat degradation is avoided.

However, although this process eliminates the problem of heat induced decomposition of the polymer, the pellets produced suffer from other disadvantages.

Most of the disadvantages stem from the inherent nature of the compaction process, more specifically the rollers and the powder feed thereto. It has been found to be very difficult to ensure that the powder feed is spread evenly across the rollers. This has the effect that control of the size of the pellets is difficult and so the size of the pellets can vary significantly.

Furthermore significant dust formation is typical for this kind of process. Additionally the pellets are commonly

friable having poor integrity and easily form dust from friction rubbing against each other, thus worsening the dust issue. Both of these issues are attributed to the poor spreading and roller compression technique.

Furthermore significant variability of the composition of the pellets and poor homogeneity of the pellets has also been observed. The issues are also believed to be associated with the poor powder distribution over the rollers. The problem of the variability of the pellet composition and the poor homogeneity of the pellets is exacerbated when the pellets are taken and used in a further processing step.

These kinds of pellets, wherein the thermoplastic polymer component of the pellets is PVOH, are used in the manufacture of water soluble PVOH pouches in extrusion / injection moulding processes. The pouches, as an example, are commonly used to contain a detergent composition for use in an automatic washing machine (laundry / dishwasher). In these applications is it vital that the pellets have high homogeneity to ensure that the pouches produced have good integrity to be stable in storage and have the expected water dissolution properties.

Pellets produced in a cold compaction process, as described above, often fail to meet the level of homogeneity required for the processing into the pouch format.

It is an object of the present invention to obviate / mitigate the problems outlined above.

According to the present invention there is provided a shaping process for making pellets of a thermoplastic extrudable resin composition comprising a thermoplastic polymer, plasticiser and optionally further additives, the plasticiser comprising a component which is solid at room temperature, wherein the process is run at a temperature above the melting point of the plasticiser and below the melting / plastification temperature of the thermoplastic polymer.

The shaping process may comprise pressing, extrusion, calendering and / or compaction. Most preferably the shaping process comprises extrusion.

The process of the present invention has been found to overcome the disadvantages associated with the prior art. Firstly as the process is operated at a temperature below the melting / plastification temperature of the thermoplastic polymer the process has been found to be extremely energy efficient. Furthermore the heat degradation of heat sensitive materials in the resin blend is dramatically reduced by the lowered process temperatures.

Additionally as the process operates above the melting point of the plasticiser (which is then allowed to cool to form the solid pellet) the pellets have been found to have a very low friability. Thus the pellets have a much lower tendency to release dust upon friction rubbing.

Furthermore as the pellets are produced at a temperature above the melting point of the plasticiser component the pellets have been found to have excellent homogeneity. More specifically both the overall composition of each pellet and the distribution of the individual components within the pellets have been found to have an high level of predictability and low variance. This is especially important when the pellets are used in a further processing step such as a second extrusion process (e.g. injection moulding) for the manufacture of an article comprising the thermoplastic polymer.

Generally the components are delivered to the shaping equipment used in the process in particulate form.

It has been found that he particle size of the raw materials used to make the pellets should be small. This has been observed to ensure high homogeneity of the pellets. The particle size of the raw materials used preferably is below 2000μm, more preferably below 1200μm, more preferably below 400μm and most preferably about 200μm.

Preferably the plasticiser is present in the composition with at least 5%, more preferably 10%, most preferably 15%.

Preferably the temperature of the material within the extruder does not exceed a temperature which is 10°C below the melting / plastification temperature of the thermoplastic polymer at any time. More preferably it does not exceed 15°C, more preferably 30°C and most preferably 45°C below the melting / plastification temperature of the

thermoplastic polymer. However, it is desired that the temperature of the material exceeds the ambient air temperature. Preferably the temperature of material within the extruder is at least 40°C, more preferably at least 45°C, and most preferably at least 50°C.

The plasticiser has to at least partially melt at the preferred operating temperature. The melting point of the plasticiser component is preferably at least 15°C, preferably at least 30°C and most preferably at least 45°C below the melting / plastification temperature of the thermoplastic polymer.

Preferably the plasticiser comprises a carbohydrate.

Carbohydrates are usually represented by the generalised formula $C_x(H_2O)_y$. The term herein also includes materials which are similar in nature like gluconic acids or amino sugars which cannot be fully represented by this formula. Other carbohydrate derivatives like sugar alcohols such as sorbitol, glucitol, mannitol, galactitol, dulcitol, xylitol, erythritol, isomaltutose and isomalt fall within this term.

Most preferred carbohydrates include the more thermally stable carbohydrates such as sorbitol, glucitol, mannitol, galactitol, dulcitol, xylitol, erythritol, isomaltutose and isomalt.

Other preferred plasticiser systems include solid fatty acid alkoxylates, fatty alcohol alkoxylates or polyalkylene glycols (such as long chain polyethylene glycol).

The plasticiser may comprise a further auxilliary component. Preferred auxilliary components include glycerin, ethylene glycol, propylene glycol, diethylene glycol, diproylene glycol, triethanol amine, diethanol amine and methyldiethyl amine.

Once the or each strand has issued from the extruder it may be permitted to cool under ambient conditions. Alternatively cooling may be assisted. One way in which this may be done is by employing a cooled metal belt onto which the or each strand issues. Another way in which this may be done is by using a cooled fluid, preferably cooled air, downstream of the extruder. Another way is by blowing a fluid, preferably air, across the or each strand. One or more of these methods may be used.

Preferably the or each strand is separated into pellets, during the manufácture.

The strands are separated into pellets preferably by cutting. However, other separation methods, for example twisting, are not ruled out. A method may be envisaged whereby the strand is twisted at intervals when still plastic, to form "sausages", which can be separated by breaking the connections, once they have become more brittle. Partial cutting or pressing or nipping or perforating (all such methods collectively called "scoring" herein) to form frangible separation webs, may also be employed, to form tablet precursors. Separation of the precursors to produce pellets may be effected during

manufacture or by the consumer, manageable lengths being provided from which the consumer breaks or twists off pellets as required. A pellet precursor may be, for example, a straight row of pellets, to be broken off as needed.

The extrusion pressure may be whatever is required to carry out the process in an efficient manner. Suitably it is in excess of 3 bar (0.3 MPa), preferably in excess of 5 bar (0.5 MPa), and more preferably is preferably in excess of 8 bar (0.8 MPa). More preferably still is preferably in excess of 12 bar (1.2 MPa). Most preferably it is in excess of 40 bar (4 MPa). The extrusion pressure preferably does not exceed 100 bar (10 MPa), more preferably 60 bar (6 MPa).

Generally the pellets are for use in injection moulding processes. The injection moulding process is preferably used for the manufacture of water soluble pouches intended to contain a detergent formulation for use in an automatic washing machine or in an automatic dishwasher. Thus the pellets preferably comprising a water-soluble / water-dispersible thermoplastic polymer

In this use the advantageous properties of the pellets produced in accordance with the invention, especially the high homogeneity have been found to be particularly beneficial. It is believed that this property is most beneficial as the integrity of the injection moulded product relies upon such high homogeneity of the composition being injection moulded as otherwise the low homogeneity will be reflected in the injection moulded product. The high

homogeneity has been found to lead to predictable water solubility of injection moulded products.

Preferably the water-soluble / water-dispersible thermoplastic polymer comprises PVOH or a derivative thereof.

Other water-soluble / water-dispersible polymers may be used in the process either as an alternative or in addition to PVOH. Preferred examples include poly(vinylpyrollidone), poly(acrylic acid), poly(maleic acid), a cellulose derivative (such as a cellulose ether / hydroxypropyl methyl cellulose), poly(glycolide), poly(glycolic acid), poly(lactides), poly (lactic acid) and copolymers thereof.

Processing aids may be present in the admixture which is processed. Preferred processing aids include mono-, di-, tri-carboxylic acids / salts thereof, fatty acids such as stearic acid / salts thereof, mono-, di- or triglycerides / salts thereof, aerosil, inorganic and organic pigments.

The invention will now be illustrated with reference to the following non-limiting Examples.

Examples:

Example 1:

The pelletising process was conducted on an extruder (twin screw, ICMA S. Giorgio, Milan (dedicated to processing of plastic blends and alloys).

The extruder had the following characteristics.

Screw diameter:	35 mm
Screw length:	40 cm
Working pressure:	30 bar
Output:	5 kg/h.
Temperature zones:	6 (T1=50°C, T2=60°C, T3=T4=90°C, T5=105°C and T6 (the die) =105°C.)

The extruder was attached to a two-roll unit used as a cooling source and connected to a pellet cutter.

The following formula was fed into the extruder in powder form.

Material	%
PVOH resin	85.0
Sorbitol	11.0
Processing aids	4.0
Total	100.0

The pellets obtained were chilled to room temperature. The formula yielded solid pellets having low friability.

Example 2:

The pelletising process was conducted on a pellet press (model V3-75 from Universal Milling Technologies).

The press had the following characteristics.

Die diameter:	350 mm
Holes diameter:	2 mm
Hole length:	3 mm
Infeed cone:	45°
Space between die / rollers:	1.5 mm
Die speed:	5m/s
Motor:	30 kW
Temperature:	98-102°C

The following formulae were fed into the extruder in powder form.

Material	Formula 1	Formula 2	Formula 3
PVOH resin	81.0	87.0	85.0
Sorbitol	15.0	11.0	11.0
Processing aids	4.0	2.0	4.0
Total	100.0	100.0	100.0

The pellets obtained were chilled to room temperature. Each formula yielded solid pellets having low friability.

CLAIMS

1. A shaping process for making pellets of a thermoplastic extrudable resin composition comprising a thermoplastic polymer, plasticiser and optionally further additives, the plasticiser comprising a component which is solid at room temperature, wherein the process is run at a temperature above the melting point of the plasticiser and below the melting / plastification temperature of the thermoplastic polymer.

2. A process according to claim 1, wherein the process comprises pressing, extrusion, calendering and / or compaction.

3. A process according to claim 1 or 2, wherein the plasticiser is present in the composition in at least 5%, more preferably 10%, most preferably 15%.

4. A process according to claim 2 or 3, wherein the shaping process comprises extrusion.

5. A process according to claim 4, wherein the temperature of the material within the extruder does not exceed a temperature which is 10°C, more preferably 15°C, more preferably 30°C and most preferably 45°C below the melting / plastification temperature of the thermoplastic polymer at any time.

6. A process according to claim 4 or 5, wherein the temperature of material within the extruder is at least

40°C, more preferably at least 45°C, and most preferably at least 50°C.

7. A process according to any one of the proceeding claims, wherein the particle size of the raw materials used is below 2000µm, more preferably below 1200µm, more preferably below 400µm and most preferably about 200µm.

8. A process according to according to any one of claims 1 to 7, wherein the plasticiser comprises a carbohydrate.

9. A process according to claim 8, wherein the carbohydrate is selected from the group comprising gluconic acids, amino sugars, sugar alcohols such as sorbitol, glucitol, mannitol, galactitol, dulcitol, xylitol, erythritol, isomaltutose and isomalt.

10. A process according to claim 8, wherein the carbohydrate is selected from the group comprising sorbitol, glucitol, mannitol, galactitol, dulcitol, xylitol, erythritol, isomaltutose and isomalt.

11. A process according to any one of claims 1 to 10, wherein the thermoplastic polymer is water-soluble / water dispersible.

12. A process according to claims 11, wherein the thermoplastic polymer comprises PVOH or a derivative thereof.

13. A process according to any one of claims 1 to 12, wherein the thermoplastic polymer comprises poly(vinylpyrollidone), poly(acrylic acid), poly(maleic acid), a cellulose derivative (such as a cellulose ether / hydroxypropyl methyl cellulose), poly(glycolide), poly(glycolic acid), poly(lactides), poly (lactic acid) and copolymers thereof.

14. A process according to claim 12 or 13, wherein the pellets are for use in injection moulding processes.

15. A process according to any one of claims 12 to 14, wherein the injection moulding process is used for the manufacture of water soluble pouches intended to contain a detergent formulation for use in an automatic washing machine or in an automatic dishwasher.

ABSTRACT

PROCESS

The invention comprises a shaping process for making pellets of a thermoplastic extrudable resin composition. The resin composition comprises a thermoplastic polymer, plasticiser and optionally further additives. The plasticiser comprises a component which is solid at room temperature. The process is run at a temperature above the melting point of the plasticiser and below the melting / plastification temperature of the thermoplastic polymer.

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/GB04/005273

International filing date: 16 December 2004 (16.12.2004)

Document type: Certified copy of priority document

Document details: Country/Office: GB
Number: 0329529.2
Filing date: 19 December 2003 (19.12.2003)

Date of receipt at the International Bureau: 02 March 2005 (02.03.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)